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(54) Silicon carbide-based molded member for use in semiconductor manufacture

(57) A silicon carbide-based member for use in semiconductor manufacture when a semiconductor manufacturing element for use in manufacturing a semiconductor device which is substantially free of contamination at a high yield is produced by economic cleaning or purification within a short period of time, wherein an allowable content of vanadium is 60 ppm and/or an allowable total content of heavy metal elements of iron, nickel and chromium is 100 ppm, and, in either case, an allowable total content of alkali metal elements is 10 ppm or less.

The drawings originally filed were informal and the print here reproduced is taken from a later filed formal copy.

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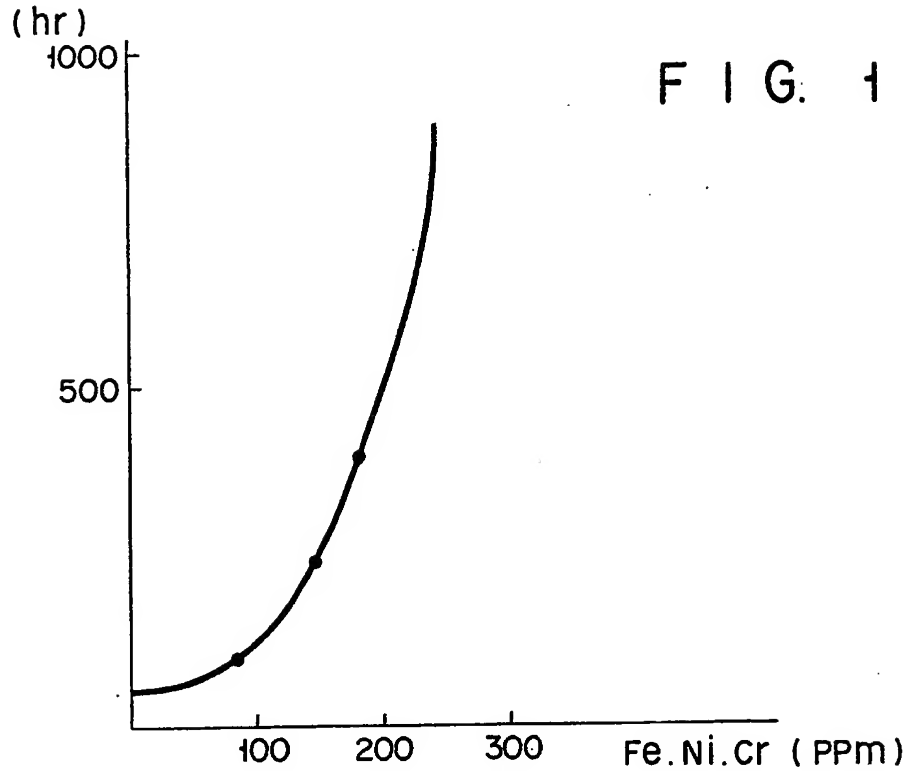

FIG. 1

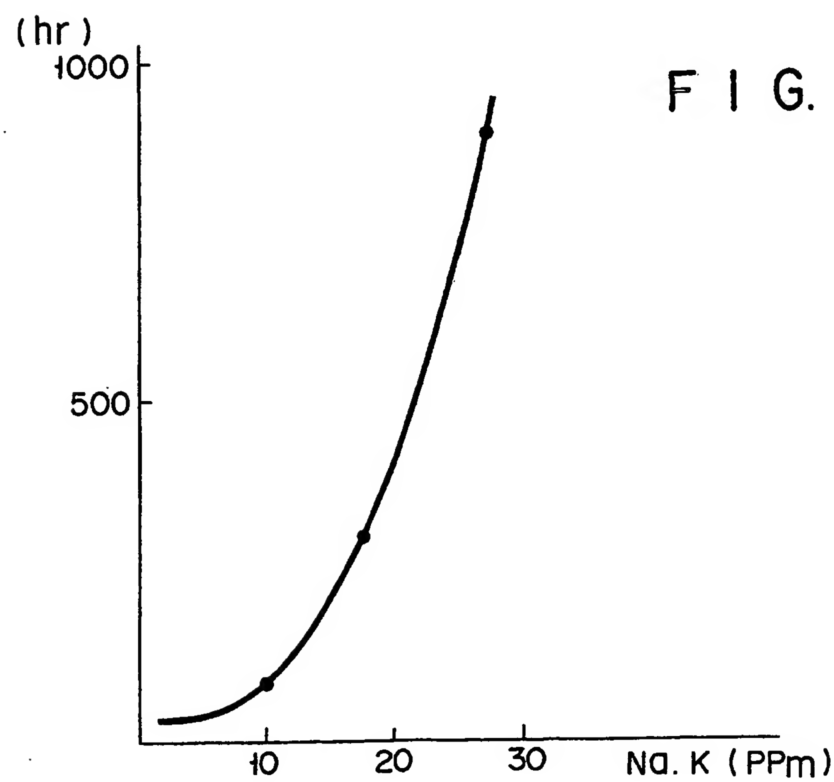

FIG. 2

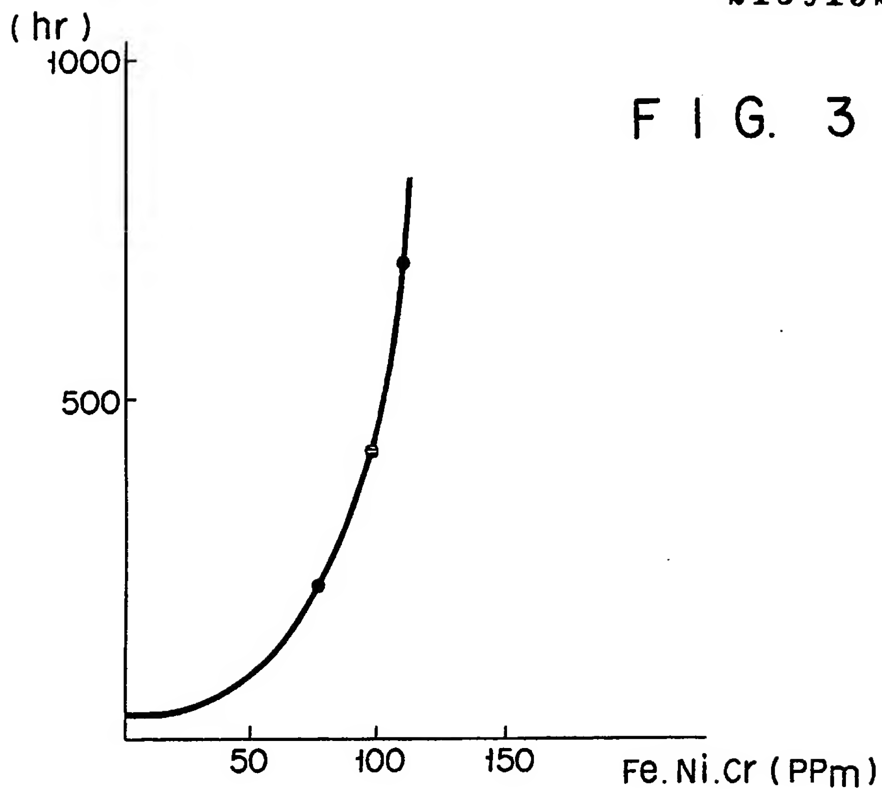

FIG. 3

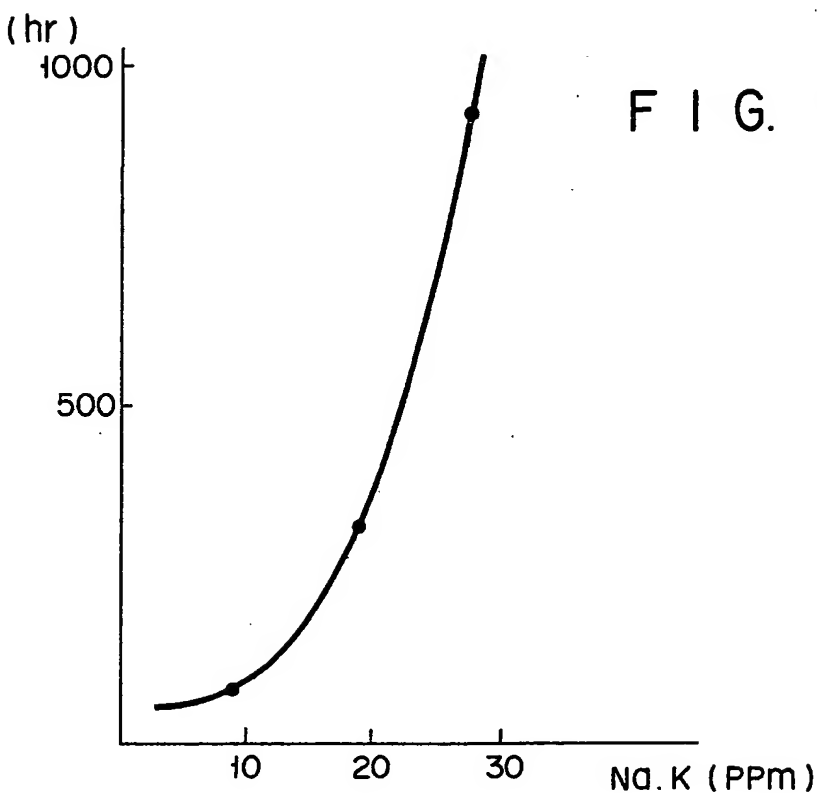

FIG. 4

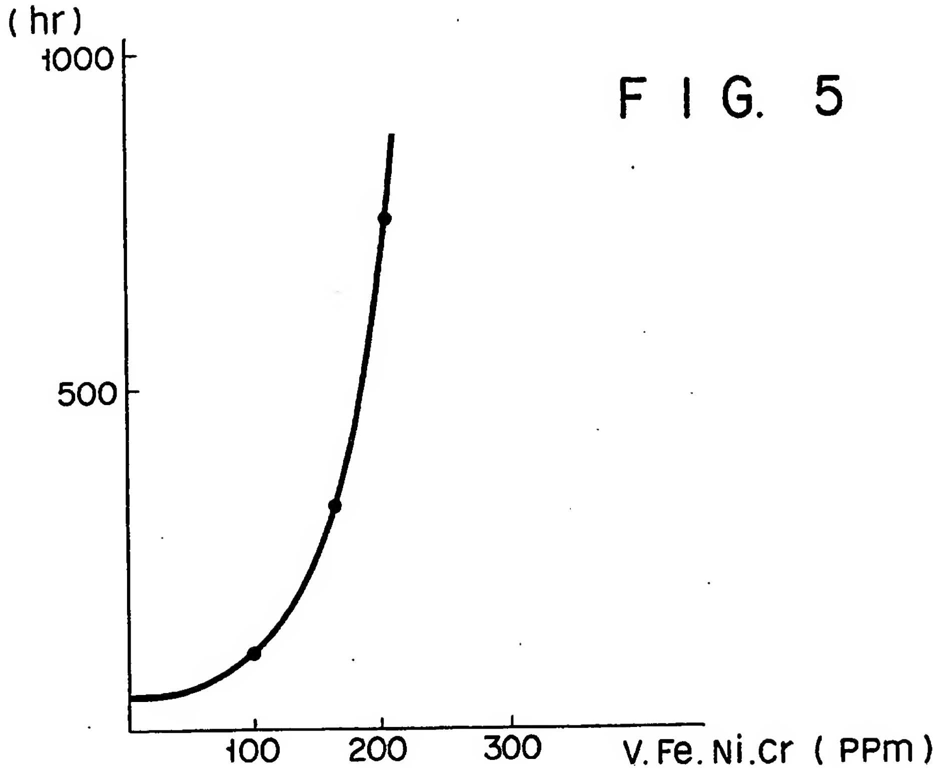

FIG. 5

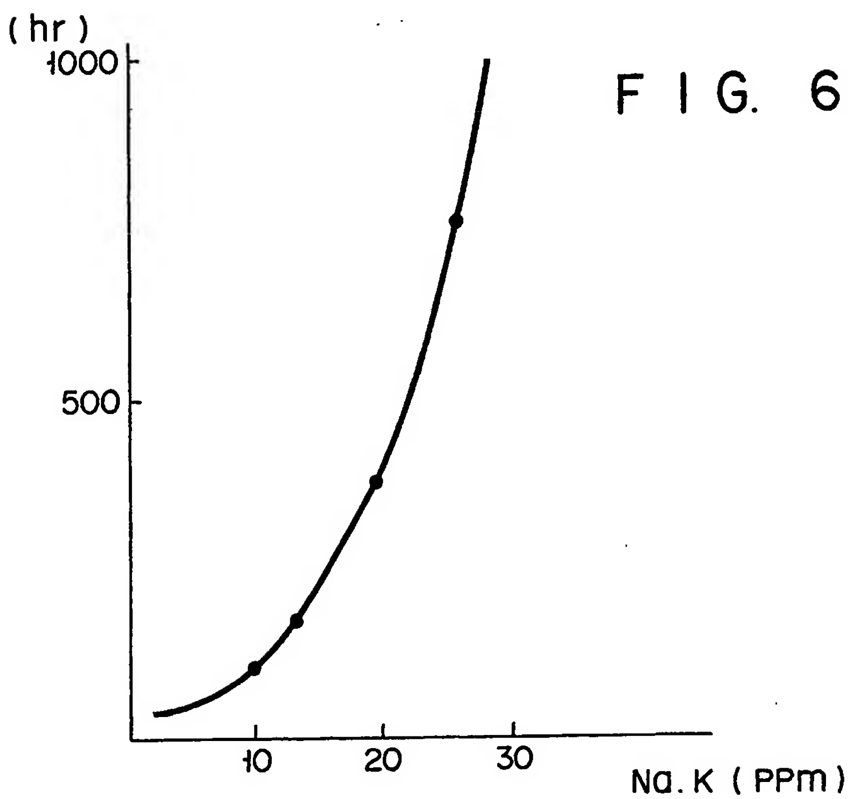

FIG. 6

SPECIFICATION

Silicon carbide-based molded member for use in semiconductor manufacture

The present invention relates to various types of silicon carbide-based molded members such as a process tube, a liner tube, and a wafer boart, and more particularly, to a silicon carbide-based molded member for use in semiconductor manufacture wherein a semiconductor device can be manufactured without being substantially contaminated, due to prior cleaning for a short period of time.

It is known to use a SiC—Si series material to form a semiconductor manufacturing member such as a process tube, a linear tube, a wafer boart and the like. Conventional SiC—Si members for this purpose are described in U.S.P. No. 3,951,587 and Japanese Patent Publication No. 55—58527. The member described in the former prior art contains a silicon carbide matrix as a major constituent which is impregnated with highly pure metal silicon so as to obtain impermeability to gases. this member is used as a constituting member of a semiconductor diffusion furnace. The member described in the latter prior art is prepared such that free silicon liberated from silicon nitride is impregnated in silicon carbide. During this impregnation process, the content of copper is kept to be less than 20 ppm and the content of an alkali metal is kept to be less than 100 ppm. This SiC—Si member is used as a SiC—Si soaking pipe which is impermeable to gases.

Such silicon carbide-based members for use in semiconductor manufacture have advantages in heat conductivity, resistance to spalling, impermeability to gases, and so on. These silicon carbide-based members are molded and purified in a predetermined manner, thus obtaining silicon carbide-based products. For purification, the silicon carbide-based molded body is heated at a temperature of about 1,300°C in an atmosphere of HCl gas. The purified silicon carbide-based product is further cleaned by pickling or by HCl gas purging, or is coated with an oxide film prior to its application. The resultant silicon carbide-based product is then used in semiconductor manufacture.

However, along with the higher packing density of semiconductor devices and an increase in diameter of silicon wafers, even if only a small amount of an impurity is present while the silicon wafer is subjected to oxidation and diffusion, the semiconductor device is contaminated to degrade the performance of the semiconductor device and decrease the yield thereof. These drawbacks cannot be ignored. In order to improve the performance of the semiconductor device and increase the yield; final-stage purification of the silicon carbide-based products used in semiconductor manufacture and cleaning immediately prior to the application of the molded products are performed more strictly than ever. However, purification and cleaning are conventionally performed such that the silicon carbide-based product is placed in an atmosphere of HCl gas at a temperature of about 1,300°C for a long period of time so as to remove the impurities. The treatment time is thus further increased, resulting in time-consuming operation. For this reason, the manufacturing cost of the semiconductor elements is increased, thus degrading the manufacturing efficiency.

Purification and cleaning are performed to clean only the surface portion of the molded products, thus an impurity component inside the products cannot be removed. An impurity diffused inside a SiC—Si molded product of the type described above is further diffused toward the surface every time annealing of the molded product is performed. In practice, the impurity diffused inside the molded product diffuses toward surface and is discharged even during cleaning, which is performed in an atmosphere of HCl gas at a high temperature. As a result, cleaning is a time-consuming process. In addition to this disadvantage, once the silicon carbide-based molded product for use in semiconductor manufacture is applied in the practical manufacture of semiconductor devices, the product is heated while the silicon wafer is subject to annealing and impurity diffusion. During these operations, the impurities diffused inside the silicon carbide-based molded product migrate toward its surface. Periodical cleaning of the semiconductor manufacturing members is prolonged, resulting in inconvenience. Therefore, those skilled in the art require a silicon carbide-based product wherein an impurity diffused inside the product will not be discharged to its surface even if the product is heated at a high temperature, so that a semiconductor device is substantially free of contamination, and cleaning of the product can be effectively performed within a short period of time.

It is a first object of the present invention to provide a semiconductor manufacturing silicon carbide-based molded member for use in manufacturing a semiconductor device which is substantially free of contamination.

It is a second object of the present invention to provide a semiconductor manufacturing silicon carbide-based molded member for use in manufacturing a semiconductor device without degrading the performance of the semiconductor device or decreasing its yield.

It is a third object of the present invention to provide a semiconductor manufacturing silicon carbide-based molded member wherein cleaning performed after molding of the silicon carbide-based material can be economically performed within a short period of time.

It is a fourth object of the present invention to provide a semiconductor manufacturing silicon-carbide molded member wherein an impurity will not be discharged therefrom, thereby substantially preventing a semiconductor device from becoming contaminated while the molded member is being used.

In order to achieve the above objects of the present invention, there is provided a semiconductor

manufacturing silicon carbide-based member wherein an allowable content of V (vanadium) is set to be 60 ppm, and/or a total allowable content of heavy metal elements, i.e., Fe, Ni and Cr, is set to be 100 ppm, and a total content of alkali metal elements is kept to be not more than 10 ppm.

According to the present invention, an impurity will not be substantially diffused or discharged from a semiconductor manufacturing silicon carbide-based molded member while the molded member is being used, thereby manufacturing a high-performance semiconductor device which does not become contaminated, and the molded member can be effectively cleaned within a short period of time.

This invention can be more fully understood from the following detailed description when taken in conjunction with the accompanying drawings, in which:

Fig. 1 is a graph showing the relationship between the total content (ppm) of heavy metals Fe, Ni and Cr contained in a soaking pipe of a silicon carbide-based material for use in semiconductor manufacture and the time during which soaking pipes having various impurity contents are purified in an atmosphere of HCl gas so that the purified pipes may serve to provide a semiconductor element having a lifetime (MOS—τ) of 200 μsec;

Fig. 2 is a graph showing the relationship between the total content (ppm) of alkali metals (Na and K) contained in a soaking pipe of the same silicon carbide-based material as that in Fig. 1 and the time during which soaking pipes having various impurity contents are purified in an atmosphere of HCl gas so that the purified pipes may serve to provide a semiconductor element having an N_{FB} value of 1×10^{11} cm^2;

Fig. 3 is a graph showing the relationship between the content (ppm) of V contained in a soaking pipe of a silicon carbide-based material which is different from those shown in Figs. 1 and 2 and the time during which soaking pipes having various impurity contents are purified in an atmosphere of HCl gas so that the purified pipes may serve to provide a semiconductor element having an etching pit density of 60/cm^2;

Fig. 4 is a graph showing the relationship between the total content (ppm) of alkali metals (Na and K) contained in a soaking pipe of the same silicon carbide-based material as that in Fig. 3 and the time during which soaking pipes having various impurity contents are purified in an atmosphere of HCl gas so that the purified pipes may serve to provide a semiconductor element having an N_{FB} value of 1×10^{11}/cm^2;

Fig. 5 is a graph showing the relationship between the total content (ppm) of V, Fe, Ni and Cr contained in a soaking pipe of a silicon carbide-based material different from those shown in Figs. 1 to 4 and the time during which soaking pipes having various impurity contents are purified in an atmosphere of HCl gas so that the purified pipes may serve to provide a semiconductor element having a lifetime (MOS—τ) of 200 μsec; and

Fig. 6 is a graph showing the relationship between the content (ppm) of alkali metals (Na and K) contained in a soaking pipe of the same silicon carbide-based materials as shown in Fig. 5 and the time during which soaking pipes having various impurity contents are purified in an atmosphere of HCl gas so that the purified pipes may serve to provide a semiconductor element having an N_{FB} value of 1×10^{11}/cm^2.

Silicon carbide-based molded members for use in semiconductor manufacture according to the present invention include all silicon carbide-based molded members that are used to manufacture a semiconductor device. Typical examples of the silicon carbide-based molded members of this type are a process tube, a liner tube, a wafer boart and a paddle. A starting material for preparing a silicon carbide-based molded member of the present invention comprises highly pure silicon carbide powder having a particle size of 40 to 200 μ. Lamp black and phenolic resin are added to the silicon carbide powder as needed. A resultant mixture is then kneaded to prepare a granulate. The granulate is then dried and is molded into a predetermined molded body such as a process tube in accordance with a known process. During the molding operation, an impurity is inevitably mixed in. In other words, silicon carbide powder which are currently used are prepared such that polycrystalline silicon carbide (called ingot) having large particles is pulverized by a stainless steel pulverizer or sieved to obtain a uniform particle size. However, when silicon carbide having a high hardness is pulverized, impurities such as Fe, Cr and the like are inevitably mixed therein in a large amount. In addition, impurities are mixed in during the screening process for adjusting the particle size. A large amount of V is contained as an impurity in carbon either used as a material or present as residual nonreacted carbon from the preparation of the silicon carbide. These impurities are sufficiently removed by repeated pickling. Pickling is generally performed in an HCl gas atmosphere at a temperature of 1,200 to 1,300°C. However, in practice, it is very difficult to completely remove the impurities unless pickling is performed for a long period of time without giving consideration to manufacturing cost.

The present inventors examined the impurities which were mixed in semiconductor manufacturing elements to degrade the performance thereof and found typical impurities (i.e., alkali, Fe, Cr, Ni and V). These impurities contained in the starting material are partially exposed on the surface of the molded body obtained by press or injection molding, but are mostly diffused inside the molded body. The molded body is heated in the atmosphere of HCl gas in the subsequent process and is purified. The primary molded bodies are controlled to have a predetermined degree of purity in accordance with required purity conditions. Thereafter, the purified molded body is impregnated with melted silicon in a

furnace, so that pores of the molded body are filled with the melted silicon. In this state, it is extremely difficult to remove the impurities diffused inside the molded body by the subsequent cleaning operation which is performed to expel the impurities to the surface of the molded body.

According to the present inventors, the silicon carbide-based molded body having pores which are not yet filled with the melted silicon can be sufficiently purified so as to control the amount of impurities to be less than an allowable limit.

The amount of impurities contained in the silicon carbide-based molded body is preferably minimized. However, removal of all impurities becomes uneconomical.

The present inventors have made extensive studies of methods of performing economic cleaning of a silicon carbide-based molded body within a short period of time, such that a semiconductor device is not substantially contaminated by impurities from the molded body.

The present inventors found that, among the impurities contained in a silicon carbide-based molded member used in semiconductor manufacture, the allowable vanadium content is at most 60 ppm and/or the total allowable heavy metal content (Fe, Ni and Cr) is at most 100 ppm, and, in either case, the total content of alkali metal elements is 10 ppm or less. In particular, the allowable content of V is more preferably 30 ppm, the total allowable content of Fe, Ni and Cr is preferably 30 ppm, and the upper limit of the alkali metal content is more preferably 7 ppm. Furthermore, in the most preferable case, the allowable content of V is 5 ppm, the total allowable content of Fe, Ni and CR is 20 ppm, and the alkali metal content is 5 ppm. When the impurity content of the silicon carbide-based molded body is below the above-mentioned value, an extremely small amount of attached around silicon carbide elements or present in the crystal particle interface may only be diffused to the surface of the molded body. Therefore, economic cleaning within a short period of time can be performed. The allowable content of each impurity according to the present invention corresponds to an allowable limit of the impurity contained in the final product as a silicon carbide-based molded body which is already purified.

In order to prepare a highly pure silicon carbide-based molded product by removing impurities therefrom, silicon carbide powder as the major material and carbon powder must be sufficiently purified prior to their application. This purification is performed in a known manner such that impurities are repeatedly removed by pickling or are removed as a low-boiling compound by heating the product in an atmosphere of hydrogen chloride gas, freon gas, or silicon tetrachloride gas. Phenolic resin is added to the highly purified starting material, and a resultant mixture is molded and hardened. The molded body has an apparent porosity of 20%. This state is very effective in sufficiently performing subsequent purification, since the impurities diffused inside the molded body can be easily removed due to the presence of a number of pores formed in the silicon carbide-based body.

As another method for preparing a porous silicon carbide-based molded body having a small amount of impurities, it is also effective to use a highly pure silicon carbon-based powder as a starting material in accordance with a method disclosed in Japanese Patent Application No. 51—35472. According to this method, a highly pure silica powder having an average particle size of 5 mm or less and a highly pure carbon powder having the same particle size as the silica powder are mixed in a graphite container, and the graphite container is moved along a tubular furnace at a temperature of 1,800 to 2,200°C so as to prepare a highly pure silicon carbide powder at this stage. The silicon powder need not be pulverized, stirred or screened and is used as the starting material per se. Therefore, the obtained silicon carbide-based molded body has a small amount of impurities. By using this starting material, a porous molded body is obtained and is then purified until the upper limit for the impurity content in the resultant molded body is reached.

Still another method for preparing a molded body having a small amount of impurities is disclosed in Japanese Patent Application No. 54—67069. According to this method, the sintering process after molding a silicon carbide body is divided into primary sintering and secondary sintering. After the primary sintering, a halogen gas or a strong acid is used to purify a resultant molded body having a high porosity. The halogen gas permeates inside the molded body as well as into the surface layer thereof, thereby sufficiently purifying the molded body and hence obtaining a highly pure silicon carbide molded body.

The resultant molded body having a low impurity content which comes close to the allowable limit of the present invention is then impregnated with highly pure silicon, thereby obtaining impermeability to gases. For this purpose, silicon is heated and melted at about 1,600°C, then the melted silicon is absorbed into the entire area of the molded body by capillary action.

When the highly pure silicon carbide-based molded body which is impermeable to gases is obtained, it is then subjected to final purification to prepare a final product. This purification is performed such that the molded body is heated in a known atmosphere of HCl gas at a high temperature. As previously described, since purification has been sufficiently performed while the molded body had a high porosity, the final purification can be completed within a short period of time. The molded body is heated and the remaining impurities are diffused in the final purification process. Even if some of the remaining impurities is discharged to the surface of the molded body, since the total amount of remaining impurities contained in the molded body is very small, the amount of impurities diffused and discharged to the surface thereof is very small. As a result, the impurities which cause

substantial contamination of the semiconductor device can be removed within a short period of time.

The performance of a semiconductor device manufactured by using a silicon carbide-based molded body used in semiconductor manufacture, e.g., a soaking furnace, can be measured as the N_{FB} (mobile ion dose in an SiO_2 film) and the lifetime (MOS—τ).

The present inventors prepared soaking pipes having various impurity contents by using silicon-impregnated molded bodies in the following manner, and the soaking pipes were examined. Highly pure silicon carbide powder which had a 99.8% purity and a particle size of 200 to 40 μ was mixed with lamp black at a ratio of 100:5 (weight ratio), and an outer percentage of 20% (by weight) of phenolic resin was added thereto. The mixture was then well kneaded. The kneaded material was granulated by an oscillator to a particle size of 500 μ, and granules were dried. The dried granules were pressed by an isostatic press to shape a pipe having an outer diameter of 120 mm, an inner diameter of 105 mm and a length of 1,500 mm. The pipe was then heated at a temperature of 200°C to cure the phenolic resin.

The resultant pipe was placed in a purified furnace in various grades so as to examine impurity contents of the pipe. The impurity contents were examined in three different combinations (i.e., heavy metal elements of Ni, Fe and Cr and alkali metal elements; V and alkali metal elements; and heavy metal elements of V, Ni, Fe, Cr and alkali metal elements). The impurity contents were measured by sampling ten different positions of the pipe which were equally spaced apart from each other in accordance with an acid extraction-atomic spectrum analysis method. The results are shown in Tables 1 to 3.

TABLE 1

(ppm)

Chemical component	Ni	Fe	Cr	Total of Ni, Fe & Cr	Na	K	Total of Na & K
Nonpurified	19	185	31	235	113	43	156
No. 1	15	139	29	183	6	2	8
No. 2	22	98	25	145	13	4	17
No. 3	6	67	12	85	18	7	25
No. 4	3	41	5	49	7	3	10
No. 5	2	18	4	24	6	3	9
No. 6	1	15	3	19	4	2	6
No. 7	1	2	1	4	1	1	2

TABLE 2

(ppm)

Chemical component	V	Na	K	Total of Na & K
Nonpurified	125	113	43	156
No. 8	112	6	3	9
No. 9	93	15	4	19
No. 10	74	21	6	27
No. 11	55	6	3	9
No. 12	27	5	2	7
No. 13	17	3	1	4
No. 14	4	2	1	3

TABLE 3

(ppm)

Chemical component	Ni	Fe	Cr	Total of Ni, Fe & Cr	V	Na	K	Total of Na & K
Nonpurified	19	185	31	235	125	113	43	156
No. 15	16	158	31	205	108	18	8	26
No. 16	14	125	23	162	83	13	5	18
No. 17	9	83	13	105	57	10	3	13
No. 18	6	74	11	91	42	8	2	10
No. 19	4	53	8	65	27	6	3	9
No. 20	5	65	8	78	8	3	2	5
No. 21	1	11	3	15	4	2	1	3
No. 22	1	10	1	12	4	1	1	2

The soaking pipes having impurities controlled as described above were dipped in silicon melted at a temperature of 1,650°C and having an impurity concentration of 1 ppb or less, so that the melted silicon was impregnated in the pores of the pipes, which then had a porosity of 1% or less.

The soaking pipes were then heated for the required time in an atmosphere of HCl gas in the furnace and were cleaned so as to substantially prevent contamination of the semiconductor elements. The characteristics of these soaking pipes were proven not to contaminate the semiconductor elements in accordance with an N_{FB} value of $1 \times 10^{11}/cm^2$ and a lifetime (MOS—τ) of 200 μsec.

The relationship between the required heating time in the atmosphere of HCl gas and various impurity contents of the soaking pipes prior to cleaning is shown in Table 4. The HCl purge times required to obtain a lifetime (MOS—τ) of 200 μsec, an N_{FB} of $1 \times 10^{11}/cm^2$ and an etching pit density of $60/cm^3$ as functions of the total contents of the impurities in the tables (i.e., the total content of Fe, Ni and Cr; the total content of alkali elements; and the content of V) are shown graphically in Figs. 1 to 6.

TABLE 4

No.	Total content of Fe, Ni, & Cr (ppm)	Total content of alkali metals (ppm)	Content of V (ppm)	1*	2*	3*
Table 1						
No. 1	183	8		400	57	
No. 2	145	17		240	290	
No. 3	85	27		100	900	
No. 4	49	10		78	81	
No. 5	24	9		59	65	
No. 6	19	6		58	46	
No. 7	4	2		54	37	
Table 2						
No. 8		9	112		61	700
No. 9		19	93		320	420
No. 10		27	74		940	230
No. 11		9	55		78	80
No. 12		7	27		59	57
No. 13		4	17		42	56
No. 14		3	4		39	53

No.	Total content of Fe, Ni, and Cr (ppm)	Total content of alkali metal (ppm)	Content of V (ppm)	1*	2*	3*
Table 3						
No. 15	205	26	108	700	750	
No. 16	162	18	83	330	380	
No. 17	105	13	57	100	180	
No. 18	91	10	42	80	100	
No. 19	65	9	27	61	70	
No. 20	28	5	8	58	59	
No. 21	15	3	4	55	55	
No. 22	12	2	4	53	49	

1*: HCl purge time required to obtain a semiconductor element having a lifetime (MOS-τ) of 200 μsec

2*: HCl purge time required to obtain a semiconductor element having an N_{FB} value of 1×10^{11} /cm^2

3*: HCl purge time required to obtain a semiconductor element having an etching pit density of 60/cm^2

As is apparent from Fig. 1, it is found that the HCl purge time becomes substantially a given short period of time required to obtain the predetermined MOS—τ value when the total content of Fe, Ni and Cr becomes 100 ppm or less. Referring to Fig. 2, it is found that the HCl purge time becomes substantially a given short period of time required to obtain the predetermined N_{FB} value when the content of alkali metals in the same silicon carbide-based molded member as shown in Fig. 1 becomes 10 ppm or less.

As is apparent from Fig. 3, it is found that the HCl purge time becomes substantially a given short period of time required to obtain the predetermined etching pit density or less when the content of V in a silicon carbide-based molded member different from those shown in Figs. 1 and 2 is 60 ppm or less. Similarly, referring to Fig. 4, it is found that the HCl purge time becomes substantially a given short period of time required to obtain the predetermined N_{FB} value when the content of alkali elements in the same silicon carbide-based molded member as shown in Fig. 3 is 10 ppm or less. As shown in Fig. 5, it is found that the HCl gas purge time becomes substantially a given short period of time required to obtain the predetermined MOS—τ value when the total content of Fe, Ni, Cr and V contained in a silicon carbide-based molded member different from those shown in Figs. 1 to 4 is 60 ppm or less. Referring to Fig. 6, it is found that the HCl purge time becomes substantially a given short period of time required to obtain the predetermined N_{FB} value when the content of alkali metal elements in the same silicon carbide-based molded member as shown in Fig. 5 is 10 ppm or less.

As is apparent from the above experiment, the silicon carbide-based materials of the present invention can substantially protect the semiconductor elements from contamination by performing economic cleaning within a minimum period of time.

EXAMPLE

Highly pure silicon carbide powder which had a 99.8% purity and a particle size of 200 to 40μ was mixed with lamp black at a ratio of 100:5 (weight ratio), and an outer percentage of 20% (by weight) of

phenolic resin was added thereto. The mixture was then well kneaded. The kneaded material was graduated by an oscillator at a particle diameter of 500 μ, and the granules were dried. The dried granules were pressed by an isostatic press to shape a process tube having an outer diameter of 120 mm, an inner diameter of 105 mm and a length of 1,500 mm. The process tube was then heated at a temperature of 210°C to dry the phenol resin. The impurity contents were as follows:

TABLE 5

(ppm)

Impurity	Fe	Cr	Ni	V	Cu	Na	K	Al	Mg
Content	189	25	15	103	125	85	31	180	83

This molded tube was placed in a sufficiently purified furnace and HCl gas was introduced into the furnace. The molded tube was heated at a temperature of 1,300°C for 30 hours. The impurity contents after purification were as follows:

TABLE 6

(ppm)

Impurity	Fe	Cr	Ni	V	Cu	Na	K	Al	Mg
Content	13	1	2	5	1	1	1	31	2

Ten positions on the tube equally spaced apart from each other were sampled and were subjected to the acid extraction-atomic spectrum analysis method.

The molded tube was impregnated with silicon melted at a temperature of 1,650°C. The molten silicon had an impurity concentration of 1 ppb or less. The melted silicon was then impregnated in the process tube, so that the process tube had a porosity of 1.3%.

The resultant process tube was then purified in an atmosphere of HCl gas at a temperature of 1,300°C for 50 hours.

CLAIMS

1. A silicon carbide-based molded member for use in semiconductor manufacture, wherein one of a content of vanadium and a total content of heavy metal elements of iron, nickel and chromium is given such that an allowable content of vanadium is 60 ppm and an allowable total content of iron, nickel and chromium is 100 ppm, and a total content of alkali metal elements is not more than 10 ppm.

2. A member according to claim 1, wherein the total allowable content of the heavy metal elements of iron, nickel and chromium is 20 ppm.

3. A member according to claim 1, wherein the allowable content of vanadium is 5 ppm.

4. A member according to claim 1, wherein the allowable total content of the alkali metal elements is 5 ppm.

5. A silicon carbide-based molded member for use in semiconductor manufacture, wherein an allowable content of vanadium is 60 ppm, an allowable total content of heavy metal elements of iron, nickel and chromium is 100 ppm, and an allowable total content of alkali metal elements is not more than 10 ppm.

6. A member according to claim 5, wherein the allowable content of vanadium is 5 ppm, the allowable total content of heavy metal elements of iron, nickel and chromium is 20 ppm, and the allowable total content of alkali metal elements is 5 ppm.

7. A member according to claim 1, wherein said silicon carbide-based molded member comprises a process tube.

8. A member according to claim 5, wherein said silicon carbide-based molded member comprises a process tube.

9. A silicon carbide-based molded member for use in semiconductor manufacture, substantially as hereinbefore described with reference to the accompanying drawings.

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